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EXAMINATION OF SAMPLE OF GROUT AFTER 63 YEARS EXPOSURE UNDERGRO--ETC(U)
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MISCELLANEOUS PAPER SL-81-6
REPORT NO. ONWI-248

EXAMINATION OF SAMPLE OF GROUT AFTER 63 YEARS EXPOSURE UNDERGROUND

by

Jay E. Rhoderick

Structures Laboratory
U. S. Army Engineer Waterways Experiment Station
P. O. Box 631, Vicksburg, Miss. 39180

May 1981

Final Report

Approved For Public Release; Distribution Unlimited

Prepared for Office of Nuclear Waste Isolation
Battelle Memorial Institute
Columbus, Ohio 43201

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

During an investigation of the Troy Lock and Dam, New York, a core was recovered that contained part of a metal anchor that had been grouted into foundation rock. Since this grout was about 63 years old and had presumably been continuously below the water table, it provided an opportunity to study the effect of this environment for this period of time on the phase composition and microstructure of this grout.

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20. ABSTRACT (Continued)

The phase composition of the grout was studied by X-ray diffraction; its microstructure was studied by scanning electron microscopy.

It was found that the grout had a normal composition and microstructure; the environmental conditions had not had a significant effect on either composition or microstructure. The original water content had been fairly high as would be expected.

Preface

A sample of a proprietary pozzolanic product was examined in connection with work being done under U. S. Department of Energy Contract No. DE-AI97-81ET46633, dated 5 November 1980, subject: "Investigation of Composition and Properties of Cementitious Mixtures for Boreholes and Shafts."

Mr. Floyd L. Burns of the Projects Management Division, Office of Nuclear Waste Isolation, Battelle Memorial Institute, Columbus, Ohio, was Project Manager.

The work was done in the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station (WES) under the direction of Mrs. Katharine Mather, Project Leader. Mr. John M. Scanlon, Jr., was Chief of the Concrete Technology Division, which provided scientists for this work. Mr. Bryant Mather was Chief of the Structures Laboratory. This report was prepared by Mr. Alan D. Buck.

Contents

EXAMINATION OF SAMPLE OF GROUT AFTER 63 YEARS EXPOSURE UNDERGROUND

Background

1. Two major concerns with plugging boreholes to isolate nuclear waste repositories are (a) the plug-host rock contact* or plug-steel casing contact** and (b) the longevity of the plug in an adverse environment.† The sample described in this report provided an opportunity to study a grout that was in contact with foundation rock as well as a steel anchor bolt while being located below the groundwater table for 63 years.

Sample Description

2. A sample of grout estimated to be 63 years old was examined to determine its phase composition and microstructure and whether it had been affected adversely by exposure conditions during this period of service. The sample was obtained in 1978 as part of a condition survey of Troy Lock and Dam†† for the U. S. Army Engineer District, New York, by the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station. The lock and dam were built in 1915 on the Hudson River in New York.

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* Office of Nuclear Waste Isolation. 1979. "Development of Plan and Approach for Borehole Plugging Field Testing," Report No. ONWI-3, Battelle Memorial Institute, Columbus, Ohio.

** U. S. Nuclear Regulatory Commission. 1980. "Information Base for Waste Repository Design; Borehole and Shaft Sealing," NUREG/CR-0495, Volume 1, Washington, D. C.

† Office of Nuclear Waste Isolation. 1980. "Repository Sealing: Evaluation of Materials Research Objectives and Requirements - 1980," Report No. ONWI-108 (preprint), Battelle Memorial Institute, Columbus, Ohio.

†† Pace, C. E., Campbell, R., and Wong, S. 1981 "Engineering Condition Survey and Evaluation of Troy Lock and Dam, Hudson River, New York; Evaluation and Rehabilitation," Miscellaneous Paper C-78-6, Report 2, U. S. Army Engineer Waterways Experiment Station, Vicksburg, Miss.

3. The grout was part of material recovered from core D-33 drilled through monolith No. 33 into the foundation rock. The core intersected an anchor bolt surrounded by grout embedded in the foundation rock at an elevation of 3.7 m (12 ft) below mean sea level.

Test Procedure

4. A piece of the grout was removed and ground to pass a 45-µm (No. 325) sieve. The ground grout was examined by X-ray diffraction as a tightly packed powder under a vapor hood containing a beaker of hot barium hydroxide solution and a static nitrogen atmosphere.

5. Another piece of grout was removed and, after preparation, was examined using a scanning electron microscope (SEM). The grout was dried in a vacuum dessicator using a vacuum pump at 60°C (140°F) for 16 hours. A fresh fracture surface was made on the dried sample, and the piece was mounted on an SEM stub. A conductive coating was applied using a vacuum evaporator. The conductive coating consisted of a layer of carbon about 5 nm thick and a layer of gold-palladium alloy about 15 nm thick. The sample was then examined using the SEM.

Results

6. Visual inspections showed that the bond of grout to the metal anchor was good and that the grout was hard and appeared to be in good condition (see Figure 74 in Pace, Campbell, and Wong 1981).

7. X-ray diffraction examination showed the grout to consist of calcium hydroxide (CH), calcite, tetracalcium aluminate hemicarbonate-12-hydrate (hemicarboaluminate) ($C_4A\bar{C}_{0.5}H_{12}$), and tetracalcium aluminate carbonate-11-hydrate (monocarboaluminate) ($C_4A\bar{C}H_{11}$), with traces of ettringite, tetracalcium aluminate monosulfate-12-hydrate ($C_4A\bar{S}H_{12}$), hydrogarnet, and unhydrated aluminoferrite as crystalline phases. Calcium silicate hydrate (CSH) was also present but was not well defined by X-ray diffraction since it is usually only poorly crystalline. This is a normal composition for mature grout that had probably carbonated

during exposure to laboratory air for a year or more before it was examined. The presence of a little unhydrated cement as aluminoferrite is common in old portland cement paste and grout.

8. Twenty scanning micrographs were made of the piece of grout that was prepared for SEM examination. Five of these were selected and are shown in Figures 1-5. They represent three areas in the piece of grout. Figure 1 (60X) shows a large void partially filled with crystals. Figures 2-4 are different magnifications of one area (440X to 17,600X) and show typical microstructural features of the grout. Figure 5 is from the third area and may show carboaluminate crystals. The micrographs indicated two specific findings:

a. The microstructure was normal for hydrated portland cement. There was no sign of detrimental change with exposure in service under the lock wall for 63 years.

b. Comparison of the representative microstructure and void space in Figure 3 with the representative dense microstructure of 1-year-old hydrated portland cement with a low water content (see Figure 4 in Mather et al.*) indicates that this grout had a fairly high water content. This is also indicated by the presence of voids such as the one shown in Figure 1. It is to be expected that grout would have a higher water content in order for it to be placed in 1915, before the use of chemical admixtures that reduce water demand at equal consistency. No data are available on the mixture proportions of the grout made in 1915.

Conclusions

9. The 63-year-old grout has a normal phase composition and microstructure.

10. The exposure in a hole in rock, adjacent to steel, under a lock wall in the Hudson River for this length of time has not appreciably changed or harmed this grout.

11. The water content of the original mixture was fairly high.

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* Mather, K., Burkes, J. P., Wong, G. S., and Reinhold, R. E. 1978. "Effects of Accelerated Curing on Hydration Products of Cement and Cement-Fly Ash Pastes," Miscellaneous Paper C-78-11, U. S. Army Engineer Waterways Experiment Station, Vicksburg, Miss.

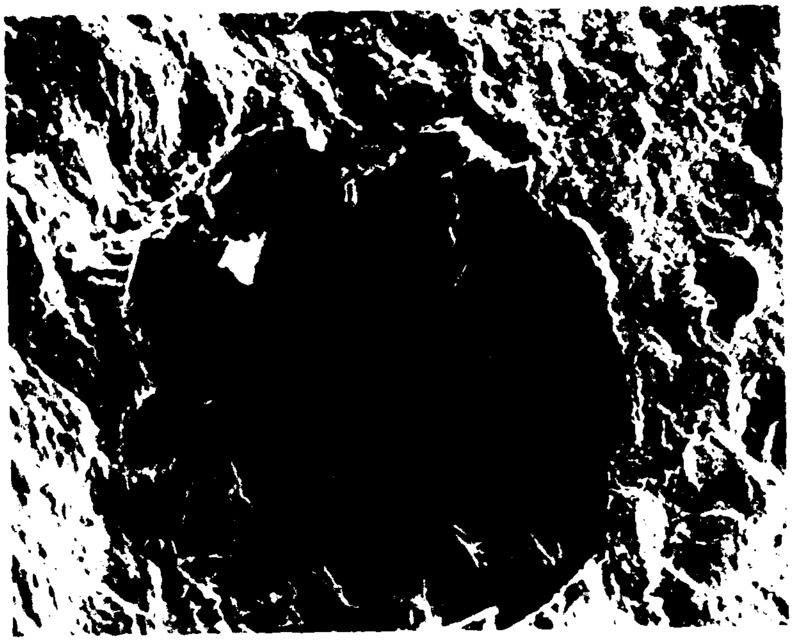

Figure 1. Needles of ettringite and crystalline plates that are probably calcium hydroxide inside a void (micrograph 050880-4, X60)

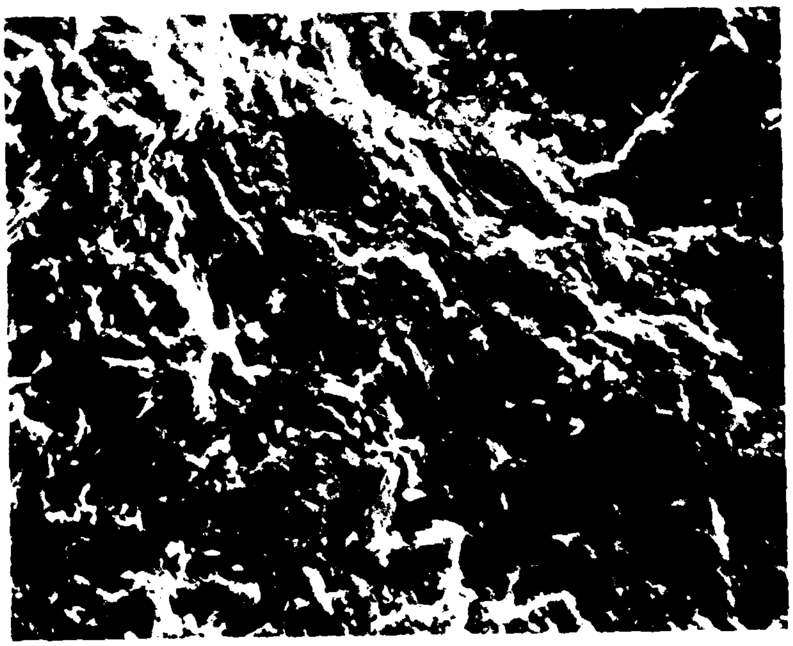

Figure 2. Typical microstructure of the grout (micrograph 050880-19, X440)

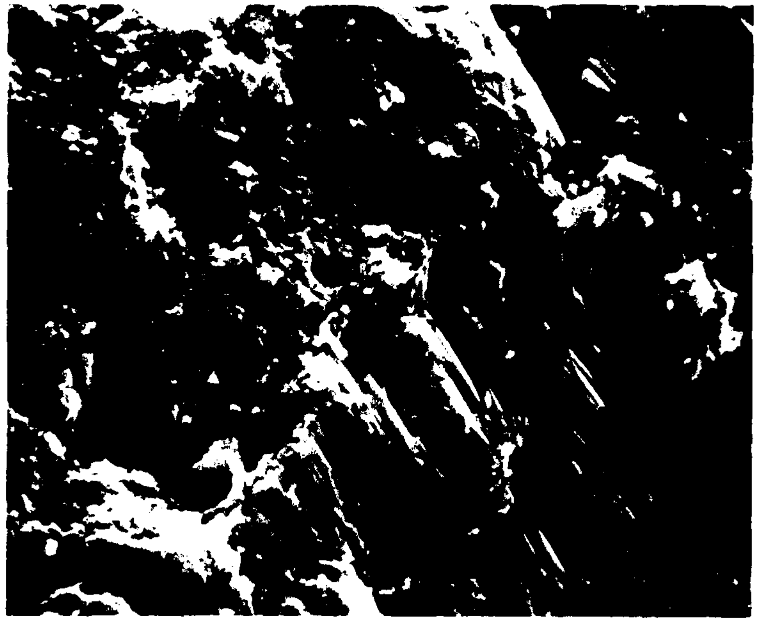

Figure 3. An enlargement of the central portion of Figure 2 showing massive calcium hydroxide (lower right) and CSH (micrograph 050880-17, X1760)

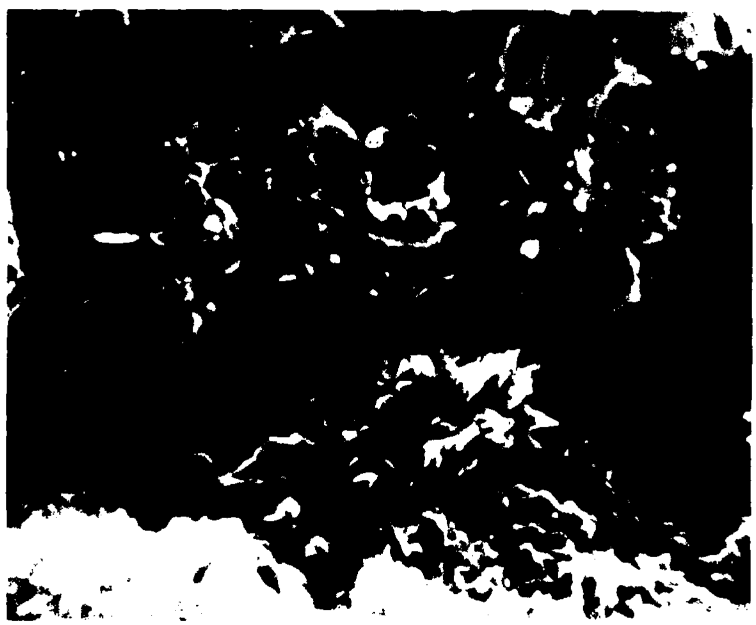

Figure 4. An enlargement of the upper center portion of Figure 3. CSH with some larger plates of calcium hydroxide (micrograph 050880-14, X17,600)

Figure 5. Narrow platelets that were sheared when the sample was split. These may be either $C_4A\bar{C}_{0.5}H_{12}$ or $C_4A\bar{C}H_{11}$ or both. The dense material is CSH (micrograph 050880-6, X3700)

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SUPPLEMENTARY

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DEPARTMENT OF THE ARMY
WATERWAYS EXPERIMENT STATION, CORPS OF ENGINEERS
P. O. BOX 631
VICKSBURG, MISSISSIPPI 39180

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14 January 1981

Errata Sheet

No. 1

EXAMINATION OF SAMPLE OF GROUT AFTER
63 YEARS EXPOSURE UNDERGROUND

Miscellaneous Paper SL-81-6
Report No. ONWI-248

May 1981

Page 1, Preface: Change the last sentence in the third paragraph to read

This report was prepared by Mr. Jay E. Rhoderick.

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